PROJECT NUMBER: 1752
PROJECT TITLE: Optical Spectroscopy of Tobacco and Smoke
PROJECT LEADER: J. O. Lephardt
PERIOD COVERED: July, 1988

I. TANDEM MASS SPECTROMETER

A. **Objective**: To establish a tandem mass spectrometry facility for R&D.

B. **Results**: The project is in a paperwork and approvals phase. The recommendations of the committee of analytical chemists involved in the project have been formalized in both written form and oral presentation to management. The paperwork is at Finance in preparation for submission to New York management.

II. CURIE POINT PYROLYSIS

A. **Objective**: To complete a project evaluating menthol release compounds and to train additional personnel in the use of the equipment.

B. **Results**: Compounds CR-2709, CR-2710, CR-2756, CR-2757, and CR-2758 were evaluated for R. Izac by Curie Point Pyrolysis - GC/MS at both 315 and 590 C.

C. **Conclusions**: Semi-quantitative data on the amount of menthol and the relative ratio of menthol to menthenes generated under pyrolysis conditions were obtained for the series of compounds. This data has been tabulated in a memo to R. Izac.

D. **Plans**: M. Buckner and N. Jensen, who received training in use of the instrumentation performing this analysis, will continue work with Curie point analysis of other samples.

III. ASHTRAY ODOR

A. **Objective**: Identification of chemical contributors to ashtray odor.

B. **Results**: The odor data for the four fractions isolated by preparative gc are not yet available. The odor distribution in the third fraction, thought to be most characteristic, was first investigated by fraction collection using retention time increments of 2 minutes. The stale, earthy and roasted notes were distributed mostly in the front sections, while the smoky and sharp notes were accumulated in the later portion. Therefore, the first 2 minute window was fraction-collected and analyzed by GC/MS with odor sniffing of the gc effluents. Mass spectral identification was focused only on those peaks with odors. Twenty-nine peaks out of about 60 peaks were detected with odors such as stale, nutty, burn leaf, and green. Most of the 29 peaks

were mixtures of at least two overlapping components, and were dominated by mass fragments characteristic of aromatic compounds - such as isopropylmethylbenzenes and ethyldimethylbenzenes - which can not explain the detected odor. Further fractionation was performed using a silica gel column with hexane, methylene chloride/methanol (9:1) and methanol as elution solvents. Separated peaks with the characteristic odor were detected in the methylene chloride/methanol fraction. Due to the dilution by the elution solvent, mass spectra obtained from the fraction were very weak and contained strong interference from the background. Therefore, to eliminate the dilution effects, chromatographic heart-cutting between two columns will be necessary to separate the interferences from the odor-bearing compounds.

C. **Plans**:

1. To identify the odor-bearing compounds by using dual column heart-cutting between a nonpolar and polar column coupled with odor-sniffing and mass spectral analysis.

2. To continue the collection and analysis of the rest of the fractions eluted between 9.5 and 20 minutes.

D. **References**:

PM Notebook 8586, pp. 62-75.